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Acoustic Emissions in Borosilicate and epoxy resin composite

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Abstract

In this paper a research looking for to extend the acoustic emission (AE) technique from the evaluation of stress state of rock samples to know its composition is presented. For this purpose the rock samples were simulated by a composite made of a resin and borosilicate spheres. The epoxy resin playing the role of country rock and Borosilicate spheres represent the coarse grain. These samples were undergone to uniaxial compression test and the AE signals were recorded and studied looking for the identification of each material characteristic spectrum. The spectral analysis of these recorded signals shown that it is possible to identify the characteristic spectra of each material from the full spectra of composite.

Key words: acoustic emission, frequency analysis, non homogeneous material.

1. Introduction

Most of the studies of Acoustic Emission (AE) are carried out in rock samples obtained directly of the mine. Intrusive igneous rocks, like Andesite, are formed from magma that cools and solidifies surrounding pre-existing rock (called country rock) and as a result these rocks are coarse grained and its behavior ought to be similar to a composite. In this work a study of AE phenomena was carried out in uncompressed samples build with two homogeneous materials which could simulates the behavior of Andesite rock. First of all a research to establish the possibility of material identification from acoustic emission of a composite was carried out.

In the last years acoustic emission technology has been successfully used to monitoring the fracture process from different materials [1], [2]. AE studies on rocks have produced interesting results monitoring the behavior of materials under stress by means of Kaiser effect studies [3], [4]. Looking for the possible source of failure in stressed materials, AE events localization techniques have been developed [5], [6]. These methods are engineer applications of AE phenomena and several NDT companies have implemented on-line monitoring systems. The majority of the available equipment for AE privileges the localization of AE sources or the study of Kaiser effect, these are based on resonant sensors which are designed for maximized the amplitude response of the AE signals but has an limited frequency response. Most of authors report that acoustic emission frequency range are typically between 100kHz-1MHz with central frequency band at 500kHz for metals and 150kHz for rocks [1] but any if one

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study have been developed to elucidate the nature of the characteristic frequency on those materials. Early studies developed by the author have shown that AE in Andesite rocks samples are focused 150 kHz band [7].

The purpose of this work is to explore the frequency band in which the AE phenomena occur in two known materials, Borosilicate and an epoxy resin samples. Afterwards a composite with the two materials was build the work consist of the identification of each material frequency signature and to explore the possibility to identify this signature in the AE of the composite. This is a first step in the possibility to identify in an intrusive igneous rock its components using AE experiments.

2. Material and method

The standard procedure to perform an AE test has been established [3]. An AE signal is a weak pulse produced in the early stages of the fracture process, to avoid catastrophic failure of sample it is necessary a controlled load system to exert pressure to the sample. Also, due the study is focused on frequency analysis of AE signal, a wideband ultrasonic transducer and a suitable data acquisition system are needed for detecting and recording the data.

In the research presented in this paper, samples made with a composite of borosilicate, and epoxy resin were tested under uniaxial load regimen in a hydraulic ENERPAC press of 15 ton of maximum load. The applied stress was measured with a strain gage in a load cell and the AE activity was detected with a wideband PAC D92043B sensor. The signal was conditioned in a preamplifier PAC 2/4/6 and afterwards amplified with a PAC-AE5A amplifier. The AE signal is digitizer with a NI 8-channels 3MS/s/ch DAQ PCI6133 digital board and signals are acquired with sampling frequency of 2.5MHz. Solid Vaseline is used for coupling the sensor to the sample. A schematic of the experimental system it is shown in the figure 1.

To establish the proper trigger level and proper amplification level a pencil lead break test [8] was carried out. Afterwards then the samples were tested from 0 to almost 20,68MPa in 1,38MPa intervals with a constant load rate of 0,34MPa/s. All data were analyzed by FFT based algorithms.

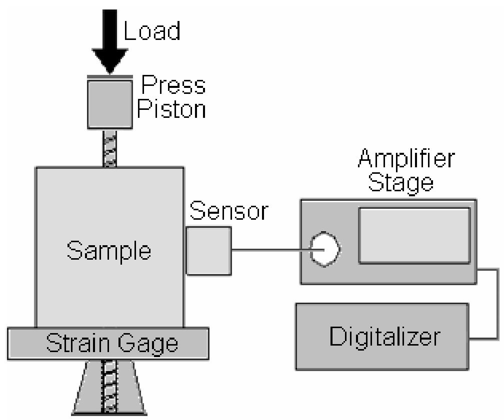

Fig.1 Schematic of the uniaxial load test performed to the samples

2.1. Sample Construction

For samples construction a composite made of 8mm in diameter borosilicate spheres and a matrix of Araldite-D epoxy were chosen. These materials were elected because it has mechanical properties different enough to expect a successful identification of it in the spectrum of AE signal generated in stress experiments. The mechanical data are shown in table 1.

Table 1 Mechanical properties of borosilicate and epoxy used to build the tested samples.

	Borosilicate	Epoxy
Fracture limit	11-12.4 MPa	>22.7 MPa
Sound velocity	2421 m/s	2597 m/s
Density	2689 kg/m^3	1139 kg/m^3

The resin has a large time to be cured, allowing the correct positioning of the spheres in the composite. In the other hand, the borosilicate sphere has a high breakage threshold and a very plastic regimen. This fact assures that the spheres start to break at higher load levels.

The samples are a rectangular prism allowing a good coupling with the sensors. The construction of the samples is made in two steps: first, a vertical aligned column made of borosilicate spheres and epoxy resin was constructed. These spheres mounted in a cylindrical mold; afterwards the mold was filled with the resin and let standing to cure.

Once the spheres column was cured it is removed from the mold and mounted in the symmetric axis of rectangular prism made of aluminum. This mold is filled with epoxy and let it cure. Figure 2(a) shows the mold to be used to construct the borosilicate spheres row. In the figure 2(b) the column of borosilicate spheres it is shown. In the figure 3(a) a schematic of final form of the test tube it is shown. In the figure 3 (b) there is a picture of finish sample.

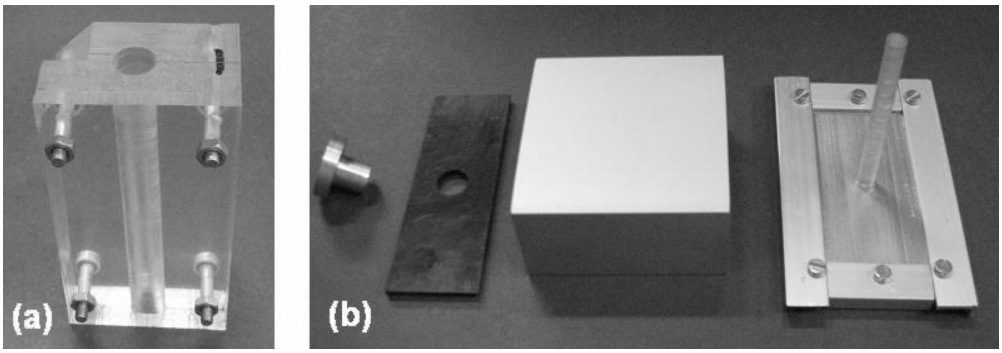

Fig.2 (a) Schematic of the samples dimension and position of the cylindrical column, (b) Photography of the column spheres.

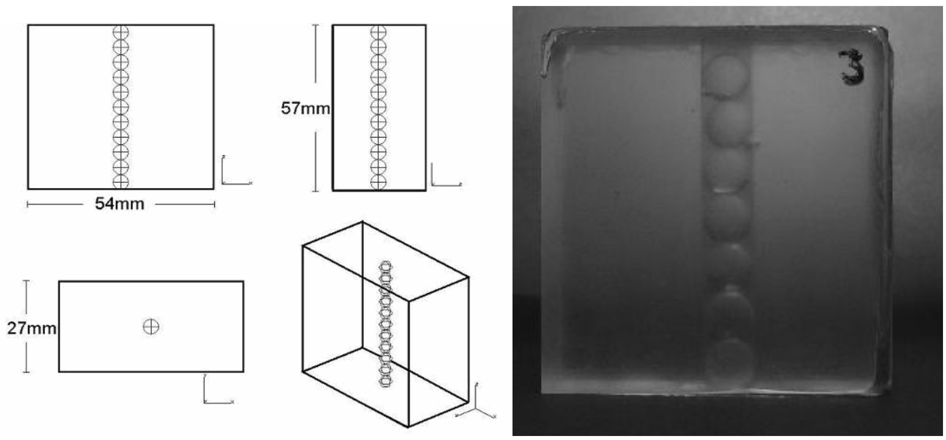

Fig.3 (a) Schematic of the samples dimension and position of the cylindrical column. (b) Photography of final form of the samples

3. Results

First experiments were carried out to identify the frequencies associated to the materials used for the composite construction. The tests consist of to stress the materials in the hydraulic press and detect the AE signals with the wide band sensor. Afterwards the signals were analyzed with signal analysis algorithms to obtain the signal spectra. Samples of single material (borosilicate and epoxy) were tested. For borosilicate samples two different geometries were used trying to avoid the influence of geometric resonances. Cylindrical samples (17mm length and 8mm diameter) and spherical samples (5mm diameter) were tested. Also rectangular epoxy samples (54mm x 57mm x 27mm) were tested. For epoxy samples mainly two frequency peaks can be identify, 140 kHz and 150kH. For borosilicates samples frequency peaks at 100 kHz, 150 kHz and 200 kHz were identify. The presence of the 150 kHz in both spectra is a sensor resonance and this value it is found in datasheet of the sensor manufacturer. There are two remarkable effects in the amplitude of the peaks. For the epoxy case the 140 kHz frequency is higher in amplitude than the resonance peak (150 kHz) so it can be easily noted despite of these frequencies are relatively closer. For borosilicate the 150 kHz peak attributed to sensor resonance is higher than the supposed material signals but, even in this case, the peak frequency of 100 kHz and 200 kHz can be resolved.

With this behavior it is expected that the material constitutive of the composite can be identified by its AE activity.

In the figure 4 the spectra with the frequency content of AE signals emitted by both material it is shown. In the figure 4a the emission of epoxy it is shown, in it can be resolved clearly the sensor frequency of 150 kHz from the material emission of 140 kHz. In the figure 4b the borosilicate AE spectrum it is shown, in this signal the difference between the material signal and the sensor resonance is clearer than in former experiment.

This experience had shown that central frequency for borosilicate and epoxy resin can be identified in a spectral analysis, in figure 4 typical spectra of these materials it is shown.

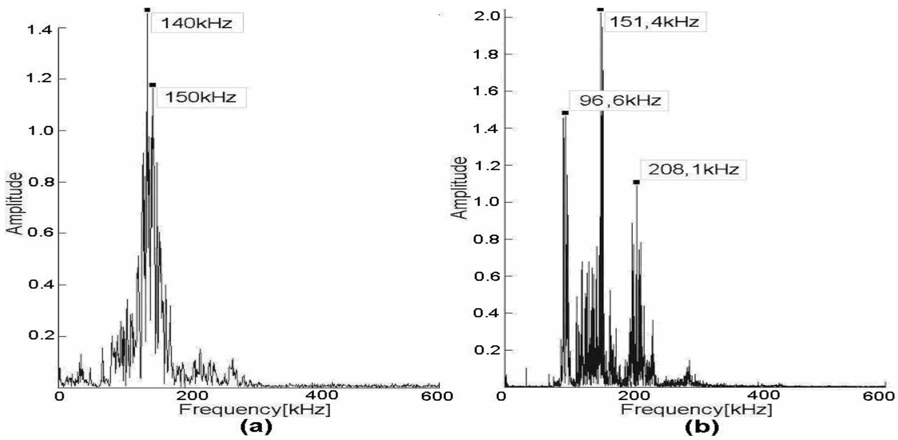

Fig.4 Frequency spectra of the materials involved. (a) Epoxy sample. (b) Borosilicate sample.

Next step is to test composite samples. The analysis of the AE data shows that the activity is presented in three different stages according to the load. The first stage goes from 0 to about 6,89Mpa. In this experiment a low AE activity was detected. The spectra content of AE events is mainly centered in the epoxy peak (140 kHz), the borosilicate characteristic frequency (200 kHz) it is also present but at low levels. A visual examination of the probes in this stage suggests that AE activity detected is mainly produced by the slip of some spheres of the cylindrical column inside the probe due to the stress applied. A characteristic AE spectrum of this stage is show in figure 5a and the slip effect is show in figure 6a.

A second stage starts from 6,89MPa to 20,68MPa. In this stage the borosilicate frequency peaks (100 kHz and 200 kHz) began to dominate the spectra also the epoxy frequency (140 kHz) seems to disappear. A visual analysis of the samples shown some of the spheres began to fracture. The fracture of the spheres starts in planes perpendicular to the load direction allowing the identification of which spheres fractures.

Finally a third stage with high AE activity start up to 20,68MPa. The spectra had shown several peaks from 100 kHz to 200 kHz and the spheres suddenly starts to collapse. The spectra are show at figure 5c and the state of the sample it is show in figure 6c.

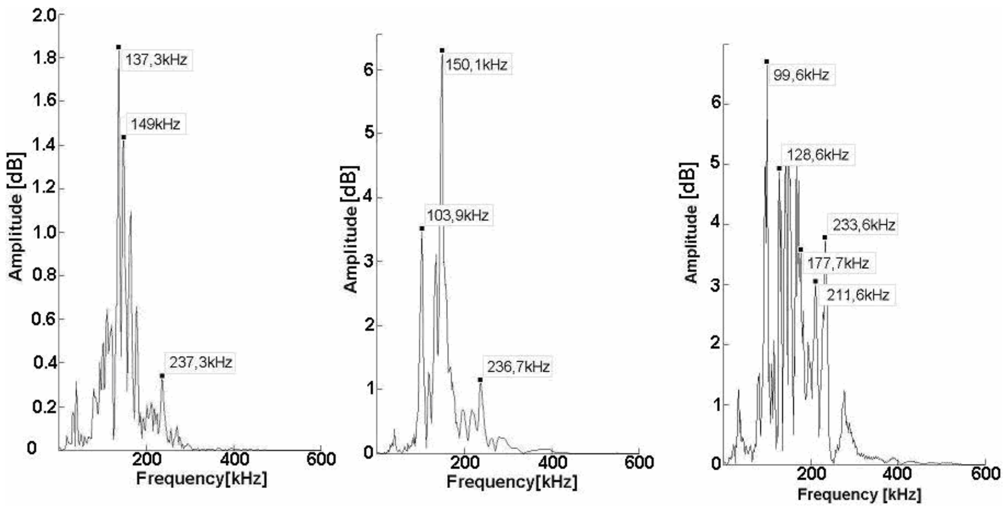

Fig.5 AE activity spectra are show for a) First stage (0-6,89MPa). b) Second stage. (6,89MPa-20,68MPa) c) Third stage (up to 20,68MPa)

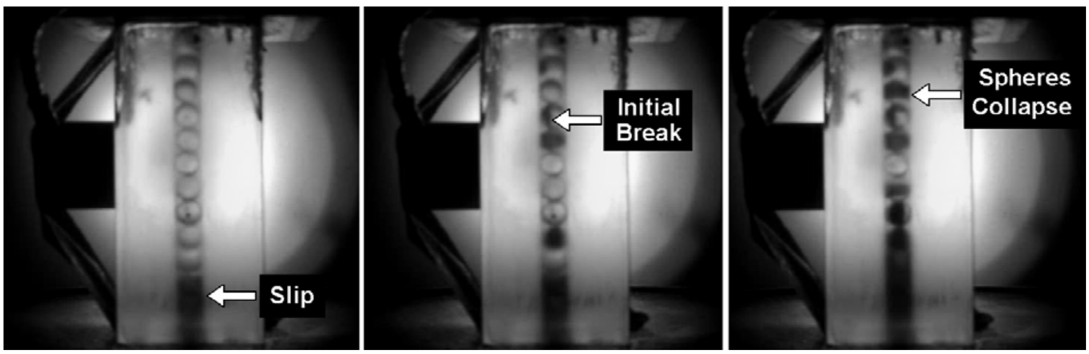

Fig.6 Photography of the state of the borosilicate spheres at the end of the load stages. a) First stage (0-6,89MPa). b) Second stage. (6,89MPa-20,68MPa) c) Third stage (up to 20,68MPa)

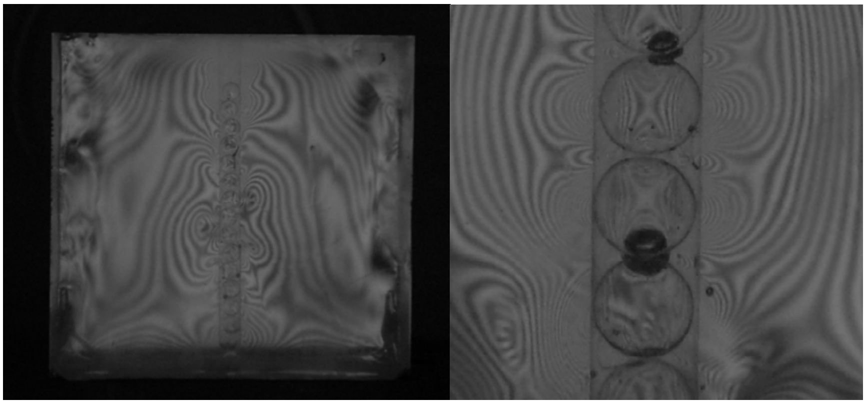

Fig.7 Polarized laser beam interference from a loaded sample showing the stress distribution. (a) Through the whole sample. (b) Close look to the junction of spheres.

4. Discussion

The spectral inspection of the signals obtained from the load test shown evidence of three stages. A first stage (0-6,89MPa) with low AE activity is detected and the mainly source is the epoxy material and the presence of the frequencies associated with this material confirms this fact. In this stage the elastic behaviour of the epoxy matrix allow it to store the energy exerted by the load and no cracks from the borosilicates spheres it is noted. Only the slips of the central cylindrical which contained the spheres is noted and this effect is the mainly source of the AE activity. The figure 6 shows the slip as a dark area surrounded the spheres.

In the second stage the epoxy release the stress to the central cylindrical and the frequency spectra shown that effect by means of the suddenly disappear of the central frequencies associated to the epoxy and the suddenly appear of the borosilicate frequency peaks. This fact is because the matrix relaxes and release the stored energy to the borosilicate spheres which start to cracks. In the figure 6 it is shows as dark lines crossing the spheres in an orientation perpendicular to the load. Finally, a last stage it is noted. Little information can be induced in this stage because the spectral shown several peaks at various frequencies but in the samples it is noted that spheres breaks and collapse. This can be shown in figure 6 by fully dark spheres. Figure 7 is an optical laser polarized beam interference of a loaded sample that shows the stress distribution on the sample at the end of the load test. It can be seen that stress distributing mainly in the surrounding area of the spheres support the fact that the epoxy matrix released the energy to the spheres.

5. Conclusion

Experiments shows that AE activity of the different materials can be identify in the AE spectra and it is possible to obtain the “signature” from different materials that can be identifies in the composite AE spectra.

Knowing the spectral characteristic of the different material that composes the sample to be tested helps to the identification of the material which is affected to the fracture process. By this way it can be identify which materials start to collapse first and the zones which this collapse occurs as it is described in the discussion section. For example, the spectra analyzed shown the epoxy peak at 140 kHz in the early stages and borosilicate peaks at 100 kHz and 200 kHz at the later stages. This is indicative that, in the test, the matrix starts to emit first than the borosilicate row.

New experimental work need to be done one devoted to locate the mains sources of the AE activity using iterative triangulation algorithm, to support the results from the visual examination of the samples. Second is to determinate the stress distribution at the sample using photo-elastometry of polarized light beam and FEM methods looking for the localization of higher stress points in the probe.

New kinds of samples should be made to prevent the slip of the central cylinder which contains the borosilicate spheres.

It will be interesting to assess the influence of AE signal amplitude in relation, for instance of the percentage content of each material.

Acknowledgment

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